

वस्त्रादि — कुछ परिरक्षकों का निर्धारण
भाग 2 एलसी-एमएस/एमएस का उपयोग करके
ट्राइक्लोसन अवशेषों की निर्धारण पद्धति
(ISO 22992-2 : 2020, संशोधित)

**Textiles — Determination of Certain
Preservatives**

**Part 2 Determination of Triclosan
Residues Method using LC-MS/MS
(ISO 22992-2 : 2020, MOD)**

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NATIONAL FOREWORD

This Indian Standard (Part 2) which is modified adoption of ISO 22992-2 : 2020 'Textiles — Determination of certain preservatives — Part 2: Determination of triclosan residues method using LC-MS/MS' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Chemical Methods of Test Sectional Committee and after approval of the Textiles Division Council.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard reference appears to certain International Standard for which Indian Standard also exist. The corresponding Indian Standard which is to be substituted in their respective places are given below along with their degree of equivalence for the edition indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 3696 Water for analytical laboratory use — Specification and test methods	IS 1070 : 2023 Reagent grade water — Specification (<i>fourth revision</i>)	Technically Equivalent

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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Indian Standard

TEXTILES — DETERMINATION OF CERTAIN PRESERVATIVES

**PART 2 DETERMINATION OF TRICLOSAN RESIDUES METHOD
USING LC-MS/MS****(ISO 22992-2 : 2020, MOD)**

WARNING — This document calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this document that the execution of its provisions is entrusted to appropriately qualified and experienced people.

1 Scope

This document specifies a method for determination of triclosan residues in textiles by high performance liquid chromatography — tandem mass spectrometry (HPLC-MS/MS).

This method is applicable to all kinds of textile products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

The triclosan is extracted from textile specimen by ultrasonic generator with methanol. After being concentrated and diluted to volume, the residue is determined by HPLC-MS/MS, quantified by external standard method.

Statistical data of interlaboratory trial is given in [Annex B](#).

5 Reagents

5.1 Water, grade 1 water specified in ISO 3696.

5.2 Methanol, HPLC grade.

5.3 **Triclosan**, CAS No. 3380-34-5, with a purity of more than 98 %.

6 Apparatus and materials

6.1 **High performance liquid chromatography — tandem mass spectrometry (HPLC-MS/MS)**, equipped with electrospray ionization source.

6.2 **Rotary vacuum evaporator**.

6.3 **Ultrasonic bath**, with controllable heating capable of maintaining a temperature of about 40 °C.

6.4 **Nitrogen blowing device**.

6.5 **Balance**, with a resolution of 0,01 g used for preparing test specimen.

6.6 **Balance**, with a resolution of 0,1 mg used for preparing standard solution.

6.7 **Glass vial**, with a capacity of 40 ml with tight closure.

6.8 **Round bottom flask**, with a capacity of 100 ml.

6.9 **Organic phase filtration membrane**, with a pore size of 0,22 µm.

7 Test procedure

7.1 Preparation of standard solution

7.1.1 Standard stock solution

Prepare 1 000 mg/l stock standard solution of the triclosan (5.3) in methanol (5.2).

7.1.2 Working solution

Prepare a working solution of the triclosan in methanol and dilute it to a series of suitable concentrations depending on test needs. Select at least five appropriate dilutions of the calibration sets to create calibration curve.

NOTE Standard stock solution is kept in the refrigerator at 0 °C to 4 °C for up to 6 months. Working solution is kept in the refrigerator at 0 °C to 4 °C for up to 3 months.

7.2 Preparation of test specimen

Prepare a representative test specimen of the sample. Cut it into small pieces with a maximum dimension less than 5 mm and mix them homogeneously. Weigh (1,00 ± 0,01) g of the pieces with a balance (6.5).

7.3 Ultrasonic wave extraction

Put the pieces into a vial with tight closure (6.7) and add 25 ml of methanol (5.2). Place the vial in an ultrasonic generator (6.3) at about 40 °C for (30 ± 2) min. Filter and transfer the extract into 100 ml flask (6.8). Add 25 ml of methanol to the residue and place the vial in the ultrasonic generator to extract the residue at about 40 °C for (30 ± 2) min. Filter and merge the extract into the flask (6.8).

Concentrate the extract with the rotary vacuum evaporator (6.2) at 40 °C to almost 1 ml. Blow slowly to nearly dry with nitrogen (6.4). Add 2 ml of methanol to dissolve the residue and then filter by filtration membrane (6.9). The filtrate is ready for determining the triclosan.

7.4 Determination of triclosan

Determine the triclosan in solution (7.3) by HPLC-MS/MS (6.1).

The test parameters by HPLC-MS/MS are given in Annex A as an example.

When the triclosan level is very low, it is necessary to increase the mass of the test pieces or concentrate the specimen liquid.

When the triclosan level is beyond the linear detector response range of the equipment, it is necessary to dilute the specimen liquid properly.

8 Blank test

Run a blank test to control contamination.

9 Calculation

The content of the triclosan is expressed by the mass ratio of the triclosan to test specimen, in mg/kg. Calculate the result by using Formula (1), and the result is rounded at two significant figures:

$$X = \frac{(C - C_0) \times V \times F}{m} \quad (1)$$

where

X is the content of the triclosan in the textile specimen, in mg/kg;

C is the concentration of the triclosan in the specimen solution, in mg/l;

C_0 is the concentration of the triclosan in the blank solution, in mg/l;

V is the final volume of the specimen solution, in ml;

F is dilution factor;

m is the mass of the test specimen, in g.

10 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 22992-2:2020;
- b) all details necessary for identification of the sample tested;
- c) the content of the triclosan, in mg/kg;
- d) any deviation from the procedure specified.

Annex A (informative)

Test parameters by HPLC-MS/MS

A.1 General

As the instrumental equipment of the laboratories can vary, no generally applicable parameters can be provided for chromatographic analyses. The following parameters have been found successfully.

A.2 HPLC test parameters

- a) Chromatographic column, C18 reversed-phase column, 150 mm (column length) × 2,1 mm (inside diameter) × 3,5 µm (particle size), or equivalent column.
- b) Mobile phase, methanol +H₂O (90:10, volume fraction).
- c) Flow rate, 0,25 ml/min.
- d) Column temperature, room temperature.
- e) Injection volume, 20,0 µl.

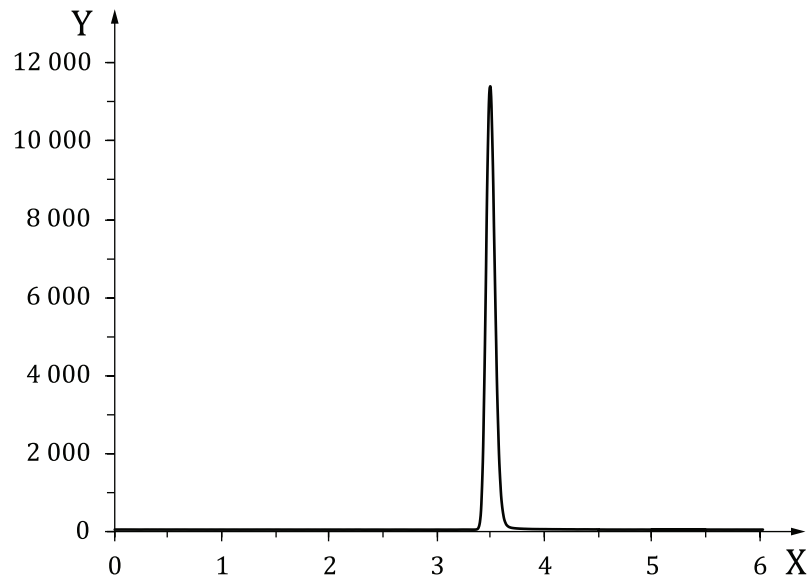
A.3 Tandem mass spectrometry test parameters

- a) Ionization mode, Electrospray ionization (ESI).
- b) Scanning mode, negative ion scanning.
- c) Detection mode, multiple reaction monitoring (MRM).
- d) Electrospray voltage, -4 500 V.
- e) Curtain gas (CUR), 30 Psi.
- f) Atomized gas (GS1), 40 Psi.
- g) Auxiliary gas (GS2), 30 Psi.
- h) Collision gas (CAD), 6,0 Psi.
- i) Ion source temperature, 550 °C.
- j) Typical transitions and detection limit shown in [Table A.1](#).

Table A.1 — Typical transitions and detection limit

Object name	Precursor ion (<i>m/z</i>)	Product ion (<i>m/z</i>)	Declustering potential (V)	Collision energy (eV)	Detection limit (mg/kg)
Triclosan	286,7	35,0 ^a	-35	-35,3	0,5
		142,1	-35	-41,5	
^a Quantitative transition					

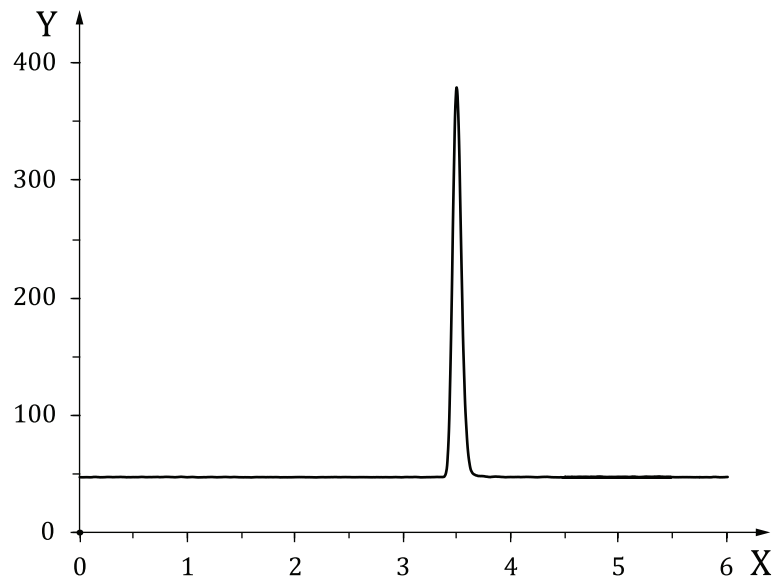
HPLC-MS/MS chromatograms are shown in [Figures A.1](#) and [A.2](#).



Key

Y abundance
X time, in min

Figure A.1 — HPLC-MS/MS chromatogram of triclosan (transitions 286,7/35,0)



Key

Y abundance
X time, in min

Figure A.2 — HPLC-MS/MS chromatogram of triclosan (transitions 286,7/142,1)

Annex B (informative)

Statistical data of interlaboratory trial

The interlaboratory trial was carried out among 9 laboratories. Three samples prepared were the fabrics separately made with cotton, polyester and silk under different concentration levels, which were cut into small pieces. In the trial, 3 parallel specimens from each sample were tested.

The statistical data are shown in [Table B.1](#). Z'-Scores of 9 laboratories were in satisfied cases ($|Z'| < 2$), except for two test results.

Table B.1 — Statistic data for triclosan content (mg/kg)

Sample No.	Fibre composition	Number of laboratories	Robust mean	S_r	r	S_R	R
A	cotton	9	33	2,0	5,0	6,0	17
B	polyester	9	90	5,0	10	15	40
C	silk	9	$2,7 \times 10^2$	5,0	15	25	70

Where
 S_r is the standard deviation of the repeatability;
 r is the limit of the repeatability;
 S_R is the standard deviation of the reproducibility;
 R is the limit of the reproducibility.

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